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DESIGN OF VACUUM CHAMBER WITH CRYOGENIC COOLING OF SAMPLES FOR BRAGG-PLANE SLOPE ERROR MEASUREMENTS*

J.W.J. Anton†, P. Pradhan, D. Shu, Yu. Shvyd'ko
Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439, USA

Abstract

Wavefront preservation is essential for numerous X-ray science applications. Research is currently underway at the Advanced Photon Source to characterize and minimize Bragg-plane slope errors in diamond crystal optics [1]. Understanding the effect of cooling the optics to cryogenic temperatures on Bragg-plane slope errors is of interest to this research. Through the use of a finite element model a custom, compact vacuum chamber with liquid nitrogen cooling of samples was designed and is being manufactured. The design process and initial results are discussed in this paper.

INTRODUCTION

Wavefront-preserving X-ray diamond crystal optics are essential for numerous applications in X-ray science [1, 2]. Perfect crystals with flat Bragg planes are a prerequisite for wavefront preservation in Bragg diffraction. However, this condition is difficult to realize in practice because of inevitable crystal imperfections. Even for practically flawless diamond crystals, internal strain of various origins, such as mounting and low-temperature cooling, can give rise to Bragg planes slope errors and significant wavefront distortions. Research is currently underway at the Advanced Photon Source to characterize and minimize Bragg-plane slope errors in diamond crystal optics [3].

One of present major goals is to develop and test schemes for mechanically-stable strain-free diamond crystal mounting with excellent heat transport to heat sinks at room and liquid nitrogen (LN) temperatures. Use of wavefront-preserving diamond crystals in x-ray Bragg diffraction at low temperatures is essential in particular for the realization of the next generation light sources of highest brilliance such as x-ray free-electron laser oscillator (XFELO) [4].

For this purpose a low-temperature compact vibration-free diamond crystal chamber is required, which could be mounted on high-precision angular goniometers in the rocking curve imaging (RCI) [3] and wavefront imaging (WFI) setups [5]. Commercially available cryostats are too bulky for this purpose.

Through the use of a finite element model a custom, compact vacuum chamber with liquid nitrogen cooling of diamond crystal samples for RCI and WFI was designed and is being manufactured. The design process and initial results are discussed in this paper.

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†anton@anl.gov

DESIGN REQUIREMENTS

To complete the the RCI and WFI studies the following design requirements were decided on:

- Rotate crystal surface ±45o
- Keep sample temp. < -185 oC long enough to conduct X-ray experiment (approx. 20 min.)
- Operate in high-vacuum environment
- Size: Compact and light as possible so it can be installed on current beamline stages

Figure 1 shows a schematic view of the chamber design. Materila with low thermal conductivity are used to mount the crystal holder and the N2 reservoir. Oxygen-free copper is used to conduct heat away from the crystal and cooled by the liquid nitrogen. Mylar sheeting was used to shield the N2 Reservoir from radiation heat loads.

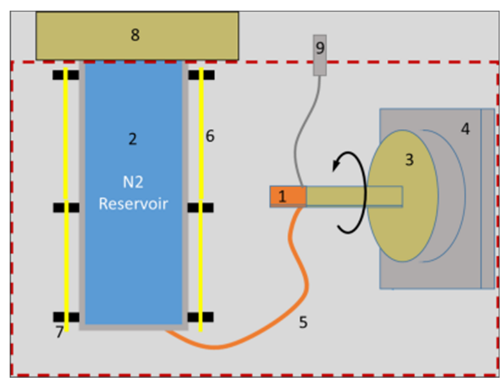

Figure 1: Schematic diagrame of the sample cryogenic cooling: 1. Crystal sample (diamond), 2. N2 reservoir, 3. crystal mount (PEEK), 4. rotation stage, 4. thermal conductor (OFHC), 5. rad. shielding (Mylar), 6. Spacer/clamps (PEEK), 7. reservoir flange (PEEK), 8. temp. sensor (RT100).

VACUUM SEAL AT CHRYOGENIT TEMPERATURES

Vacuum seal at cryogenic temperatures: Relatively large thermal contraction will happen between the N2 Reservoir (AL-6061) and the Reservoir Flange (PEEK). A vacuum seal using an indium wire allows the seal to hold even at very low temperatures. Figure 2 shows the flange mating with the N2 reservoir.

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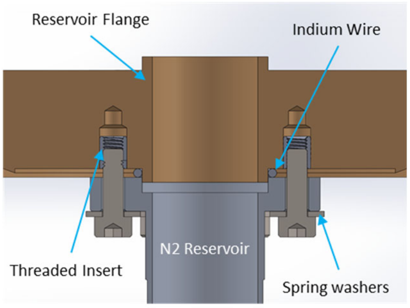

Figure 2: Reservoir flange and N2 reservoir mating with indium wire vacuum seal.

THERMAL FINITE ELEMENT ANALYSIS

A Finite Element Model was created in SolidWorks® Simulation software and a Steady State Thermal analysis was run. Conduction and Radiation heat transfer were of primary concern for this analysis. Shell elements were used for the N2 Reservoir, the Mylar Radiation Shielding, and a section of the vacuum chamber to reduce the overall number of elements in the model and thus the run time (see Fig. 3).

At the sample location the steady state temperature measured from the model is -189 ºC.

The power into the system was measered from the FE model. Length of time for all the liquid nitrogen to evaporate and the model is no longer in steady state. time the sample will stay at that this temperature (-189ºC) would be constant for approximately 16 minutes.

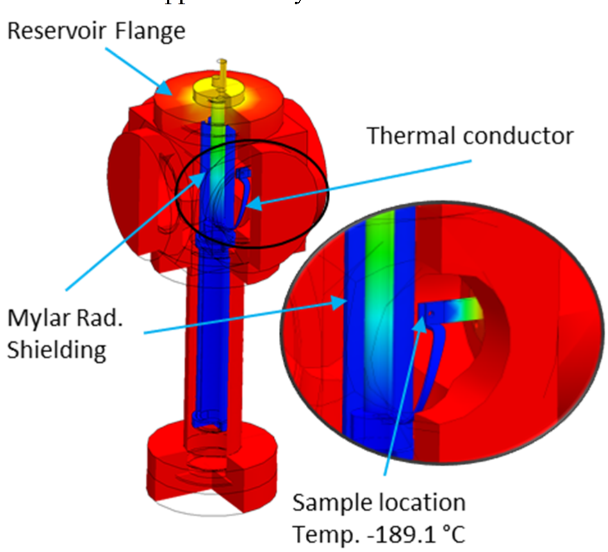

Figure 3: 3D model for thermal finite element analysis.

PROTOTYPE TESTING

From the Finite Element Analysis (FEA) results a prototype chamber was designed and built to test the coolmechanism. The prototype is shown mostly assembled in Fig. 4. An RT100 thermal sensor was fastened to the sample location to measure temperature. The chamber vacuum and cooling capabilities being tested is shown in Fig. 5.

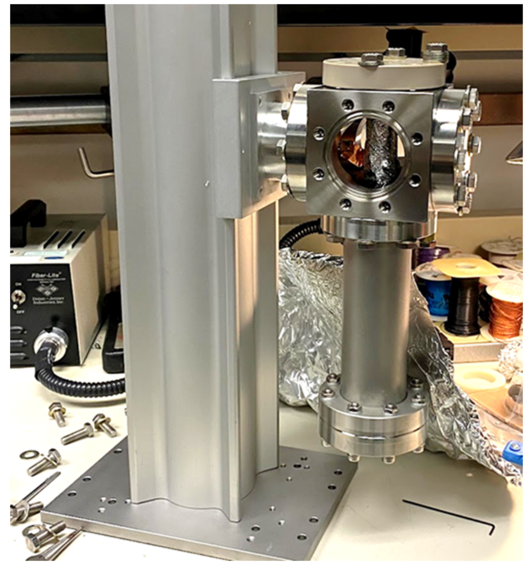

Figure 4: Photograph of the prototype sample cryogenic cooling chamber during assembly.

Figure 5: Photograph of the prototype sample cryogenic cooling chamber test setup.

As seen in Fig. 6, the prototype chamber test results were remarkably similar to the FEA results. The sample location was cooled to about -180 ºC for about 15 minutes before starting to warm. The roughly 10 ºC discrepancy between finite element analysis and prototype results is due to the thermal conductor needing to be lengthen for manufacturability of the prototype.

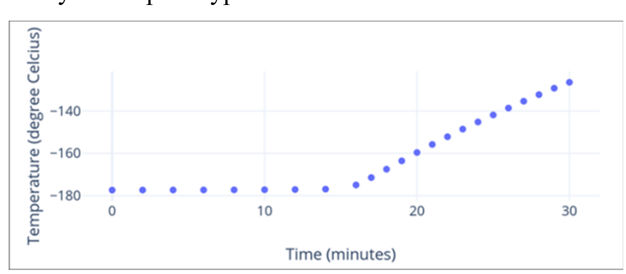

Figure 6: Preliminary test result of the prototype sample cryogenic cooling chamber.

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SUMMARY

- Using finite element analysis a compact, [approximately 120 mm x 170 mm x 300 mm] cryo-cooling vacuum chamber was designed, built, and tested.
- The sample location was cooled to near about -180 °C for 15 minutes and -160°C for more 20 minutes.
- A high vacuum environment was achieved utilizing an indium wire seal at low temperature joints high-vacuum was achieved.

These positive result lead to a final design for the chamber has being completed. The design includes a rotation stage and beryllium windows to allow the X-ray beam through to the sample. The final design is currently being manufactured. Figure 7 is a solid model of the final cryogenic cooling chamber.

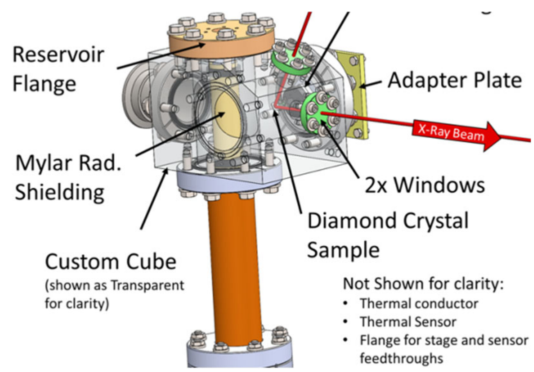

Figure 7: 3D model of the sample cryogenic cooling chamber (now being built) for Bragg-plane slope errors measurements.

ACKNOWLEDGEMENTS

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